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EFFECT OF DIFFERENT SURFACE TREATMENTS AND RESIN TYPES ON BONDING TO TRANSLUCENT ZIRCONIA

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ABSTRACT

Purpose: This in vitro study measured the adhesive properties of 10-MDP and MPE containing resin cements, using two types of zirconia surface treatments; selective infiltration etching(SIE) and airborne particle abrasion (APA) to translucent zirconia (3M, ESPE) .

Statement of Problem: Due to translucent zirconia modified composition (alumina content reduced to tenth of its weight) bonding strength to different resin cements after different surface treatments should be investigated adequately .

Materials and Methods: Sixty sintered Lava plus high translucency disks(3M,ESPE)were randomly divided into three study groups according to their surface treatment: (a) polished surface (control group); (b) air borne particle abraded (grit blasted) with 50 μm aluminum trioxide (APA); and (c) selective infiltration etching (SIE). Zirconia disks (15x2mm) were then bonded to 60 composite resin disks (8x4mm) using two different resin composite cements (Clearfil SA and Rely X). Resin-zirconia adhesion strength was evaluated using the microshear bond strength test (MSBS) after 24 hour of storage in deionized water at 37°C. One way ANOVA and Scheffé s post-hoc tests were used to analyze the data ($p<0.001$). Then fractured samples were studied under SEM and classified according to failure pattern.

Results: Different types of resin cement and surface treatment significantly influenced the MSBS ($P<0.001$). The highest mean MSBS values were recorded with MDP containing resin cement (Clearfil SA) in both SIE (26.18 ±1.12 MPa) and APA (21.67±1.34) groups. Bond strength values were reduced significantly in control group when using the two types of cements. Regarding failure pattern, MDP groups showed cohesive and MPE and control groups showed adhesive type.

Conclusion: SIE and APA in combination with 10-MDP containing resin cement established a strong durable bond to zirconia substrates than MPE groups. SIE and APA showed significant higher bond strength values than control groups.

KEY WORDS: translucent zirconia, microshear bond strength, resin composite cement, surface treatment, SIE, APA, MDP and MPE monomers.

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INTRODUCTION

Thermo-dynamic behavior of zirconia indicates that the structure of the surface grains could be manipulated by controlling both temperature and heating time [1]. Heat induced maturation (HIM) resulting in stressing the grain boundary regions by 2 short thermal cycles, but does not provide sufficient energy to allow for grain growth or cubic grain formation. Zirconia is heated to 750˚C for 2 minutes, cooled to 650˚C for 1 minute, reheated to 750˚C for an additional 1 minute and then cooled to room temperature. After this heat treatment, the grain boundaries become prestressed and can be easily penetrated by other material [1].

Yttrium concentration, which is the primary stabilizing element of zirconia used in dental restorations, was found to be higher at grain boundaries and surfaces compared with the grain interior [2].

Selective infiltration etching uses principal of heat induced maturation and grain boundary diffusion to transform the relatively smooth non-retentive surface of Y-TZP into a highly retentive surface[3]. In combination with heat induced maturation, which is used to pre-stress the grain boundary regions, these regions could be further widened by applying a thin layer of an infiltration glass over the surface of the treated zirconia. In the semi-liquid state, the molten glass infiltrates selectively between the boundaries of the surface grains and exerts surface tension and capillary forces, allowing rearrangements movements of the surface grains, and results in creation of 3 dimensional network of intergrain porosity[4].This surface treatment is selective because it involves only the surface grains in contact with the infiltration glass, the operator can control the area of zirconia that needs to be treated. For achieving a strong nano-mechanical bond with HIM/SIE treated zirconia, an optimal surface architecture would allow infiltration of adhesive resin into the created retentive features and would not result in excessive surface damage or roughness that weakens the surface [5-6].

As translucency refers to the degree to which a material allows light pass through it. In zirconia, the presence of impurities and/or structural defects can have an impact on the material translucency. Zirconia impurities can cause light absorption, obviously detracting from the material ability to allow light through [7].

Alumina is incorporated into zirconia materials in order to increase their aging stability .The drawback, however, is that alumina has a different refractive index than the rest of zirconia material, which scatters light, resulting in similar translucency drawbacks as those caused by structural defects [8].

The conventional methods of pretreating a silica based ceramic surface with hydrofluoric acid etching ,airborne particle abrasion (APA), and silica coating by APA with a silane coupling (tribochemical silica coating) seem to be ineffective for zirconia[9].

Another limitation of oxide ceramics is their relatively hydrophobic surface with a low surface free energy and a low OH-group concentration on the surface [10].

During the last fifteen years, a variety of surface treatments have been introduced to enhance the bond strength of resin composite cements to zirconia, including APA[11], tribochemical silica coating[12], ER:YAG laser irradiation[13], laser treatment[14], acidic organophosphate monomers, various functional silane monomers [44], selective infiltration etching (SIE) [16], hot acid etching and fluorination technique [17].

However, scientific evidence shows that surface treatments, such as grinding (with a bur) or APA of the yttria-stabilized tetragonal zirconia polycrystal (Y-TZP) ceramics before cementation may also create surface defects or initial flaws and sharp cracks[18].

Such imperfections can act as stress concentration foci, rendering zirconia framework susceptible to radial cracking during function [18].Such cracks generated by APA on zirconia surface may reduce its strength by about 25% when cyclically loaded. However, some researchers have demonstrated that these subtractive surface modifications may significantly increase the flexural strength of Y-TZP by inducing the transformation from the tetragonal to monoclinic phase (so called transformation toughening), which might inhibit microcrack propagation,thus increasing the strength of zirconia[19]. In addition, APA increases surface free energy, increases the surface area for bonding, and provides better wettability[20].

Zirconia is basically composed of zirconium dioxide, free of silica inherent which is responsible for surface roughness acquired during acid conditioning; contributing to improve the adhesion to cement agents. The absence of silica also makes it difficult silanization,due to affinity to silane in establishing molecular bonding with silica[21].

The purpose of this study was to evaluate the microshear bond strength of translucent zirconia luted to composite resin discs after different surface treatments using airborne particle abrasion and selective infiltration etching using MDP and MPE resin cements. Failure types of tested groups were also observed and compared under SEM .The null hypotheses of this study were:

- Luting resin containing 10-MDP with translucent zirconia does not differ in adhesion values compared to MPE containing cement.
- SIE and APA surface treatments does not differ in bond strength values of resin cement to translucent zirconia.

MATERIALS AND METHODS

Fully sintered Lava plus high translucency zirconia disks (diameter: 15mm; thickness: 2mm) were prepared by cutting zirconia milling blocks (3M,ESPE; St Paul, MN, USA), using a precision cutting instrument (Isomet 1000, buehler; Lake Bluff, IL, USA) and a diamond coated cutting disk (Diamond Wafering Blade, No 11-4276, Buehler).

The cutting procedure was precisely guided and carried out with a horizontally moving digital micrometer (IDC 1508, mitutoyo; Kawasaki, Japan). fig.1

TABLE (1) Resin cements used in the study.

Cement	Composition	Manuf.
Rely X Unicem	Methacrylated phosphoric ester(MPE), Dimethacrylate, Inorganic fillers, Fumed silica, Chemical and Photoinitiator.	3M ESPE St.Paul,Minn
Rely X Ceramic Primer	Prehydrolized silane coupling agent, alcohol, water.	
Clearfil SA	10-methacryloyloxydecyl dihydrogen phosphate (MDP), 2-hydroxyethyl methacrylate (HEMA), N-dimethyl-p-toluidine (NDPT), water.	Kuraray Medical; Tokyo, Japan.
Clearfil Ceramic Primer	MDP, ethanol, 3-(tri methoxysilyl) propyl methacrylate	
Filtek Z 250	Zirconia and silica fillers, bis-GMA, UDMA, bis-EMA, Prompt LPop: Di hema phosphate, Bis phenol A Diglycidyl ether dimethacrylate, ethyl 4-dimethylamin- Obenzoate, DL camphorquinone.	3M ESPE Seefeld, Germany

Zirconia disks were polished using silicone carbide papers starting with a 120 grit and ending with a 800 grit (Microcut, Buehler). Polishing was carried out by using a rotating metallographic polishing device (Ecomet, Buehler) under a 300gm load and water cooling. The disk shaped specimens were randomly divided into 3 groups and each group was subdivided into 2 subgroups. The first 2 groups, polished surface was used as control.

Disks in group 3 and 4 underwent airborne particle abrasion (APA) with 50μm aluminum trioxide powder (P-G 400, Harnisch & Rieth; Winterbach, Germany) under a pressure of 0.35 MPa (S-U-Alustral, Schuler-Dental; Ulm, Germany) and at a perpendicular distance of 1 cm, followed by ultrasonic cleaning in distilled water for 10 min.

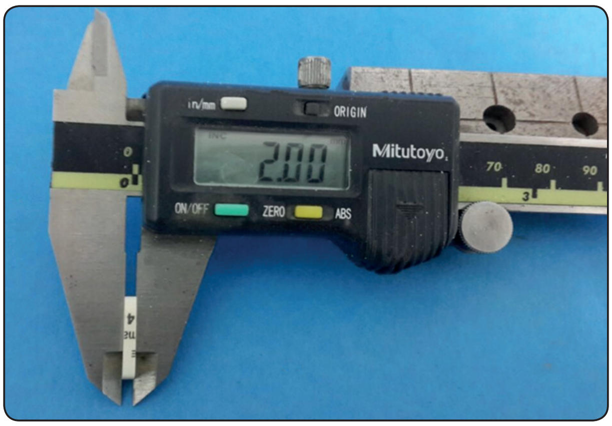


Fig. (1) Ceramic discs with standard thickness.

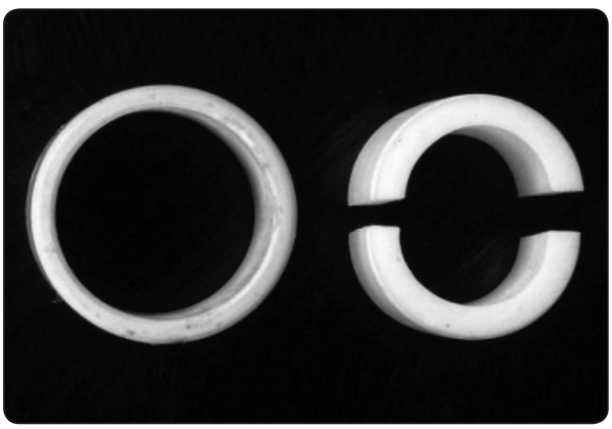


Fig. (2) Mold for composite samples fabrication.

The specimens in group 5 and 6 underwent selective infiltration etching (SIE) surface treatment as described in a previous study[1], the method employs a heat induced maturation process to pre-stress surface grain boundaries on zirconia to allow infiltration of molten glass.

Zirconia is heated to 750˚C for 2 minutes, cooled to 650˚C for 1minute, reheated to 750˚C for an additional 1 minute and then cooled to room temperature. After this heat treatment, the grain boundaries become pre-stressed and can be easily infiltrated by other materials.

After cooling to room temperature, the glass is then etched away using 5% hydrofluoric acid for 30 min. This creates a rough surface topography with deep grooves at zirconia grain boundaries, allowing nanomechanical interlocking of resin composite cement. A total of 120 resin composite (Filtek Z 250, shade A 2, 3 M,ESPE; St Paul, MN, USA) disks measuring 8.0 mm in diameter and 4mm in thickness were prepared by injecting the composite resin into a mold (Fig.2) and light polymerized for 20 sec each from the top and bottom (Elipar Free Light 2, 3M ESPE).

Luting of samples

Two adhesive resin composite cements (Table 1) Clearfil SA containing MDP (Kuraray; Tokyo, Japan) and Rely X cement containing MPE (3M, ESPE) were applied after surface treatments according to manufacturers recommendations.

Each resin cement was applied to the surface of resin composite disk using the auto mix tip; the disk was seated on the pretreated zirconia disk surface using jig loaded with a 50 N force for 60s. Excess cement was carefully wipped off.

Finally, the adhesive cement was light cured for 60s.

All specimens were stored in (deionized) water at 37˚C for 24 hours before testing.

Microshear Bond Strength Test:

Zirconia disks were fixed between 2 steel plates using a travel stage micrometer (Mitutoyo). By pushing zirconia disk downward at a cross head speed of 1.0mm/min, a shear force was applied to each bonded interface until failure occurred [(22)]. Great care was taken to properly align the specimens so that the bonded interfaces parallel to the direction of the load.

Fractured zirconia surfaces were examined after MSBS testing under a light microscope at 50 X magnifications then under a scanning electron microscope at 2000 X magnification (XL20, Philips; Eindhoven; Netherlands). Specimens were sputter coated for 2 min (5150 B sputter coater, Edwards; Crawly, UK) with fine gold powder and the failure modes were classified as: adhesive failure between cement and zirconia; cohesive failure in resin cement; mixed or adhesive /cohesive failure in resin cement (fig.3-4).

Statistical methods

SPSS version 22.0 was used for data management. Mean and standard deviation described microshear bond strength. One way ANOVA made comparisons between groups and Scheffee test made pairwise comparisons. Fisher exact test compared independent variables.

RESULTS

Data from the MSBS tests are summarized in table 2. *a*nd graph. 1. One way ANOVA showed the effect of surface treatment and type of cement $p<0.001$. The interaction between surface treatment and resin type were all significant. The Scheffe's tests showed a significant difference in bond strength values between the control group and the APA as well as SIE surface treatment group ($p<0.001$), whereas there were no significant differences between the SIE and APA groups.

Regarding the type of cements, Clearfil revealed a significantly higher MSBS value than Rely X ($p<0.001$).

Analysis of tested samples, exposed zirconia surface following fracture predominantly demonstated adhesive failure when non –MDP containing cement were used. In contrast, MDP containing Clearfil resulted in predominantly cohesive failure in the resin cement.

For the control group, the failure was always adhesive between the resin cement and zirconia (graph.2).

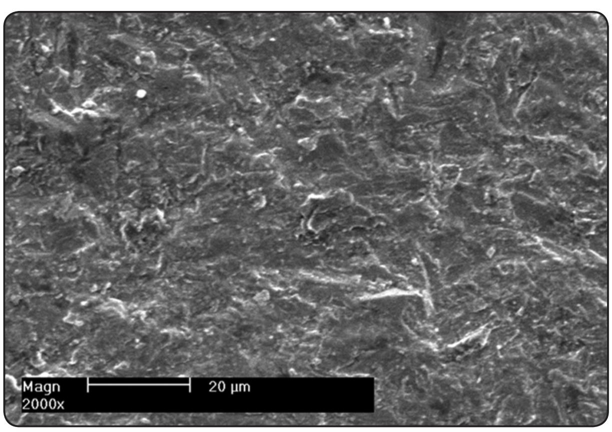


Fig. (3) Debonded translucent zirconia surface. SEM:2000 x magnification.

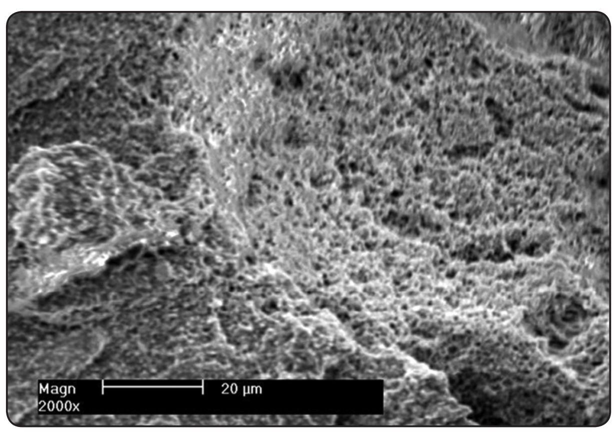


Fig. (4) Cohesive failure mode in (SIE-MDP) group. SEM:2000 x magnification.

TABLE (2) Mean Microshear bond strength (MPa) in different groups

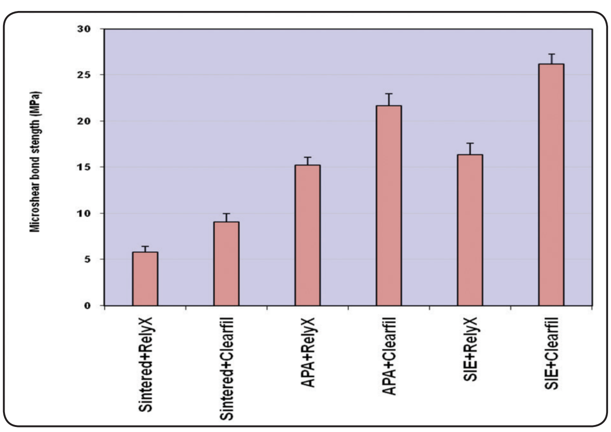
Study groups	Mean	SD	Median	Minimum	Maximum
Sintered+RelyX	5.81 (e)	0.63	5.75	4.90	6.70
Sintered+Clearfil	9.09 (d)	0.88	9.15	7.80	10.20
APA+RelyX	15.26 (c)	0.82	15.25	14.30	16.80
APA+Clearfil	21.67 (b)	1.34	21.80	20.00	24.20
SIE+RelyX	16.35 (c)	1.26	16.80	14.10	17.70
SIE+Clearfil	26.18 (a)	1.12	25.90	24.70	27.80

p value < 0.001, groups sharing same letter are not significantly different

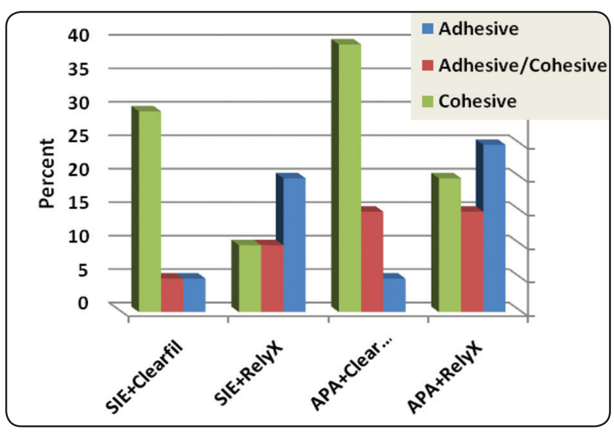
TABLE (3) Comparison of failure types in test groups

Test groups		Failure type					
		Adhesive		Adhesive/cohesive		Cohesive	
		Count	%	Count	%	Count	%
Resin cement	SIE Clearfil	1	5.0%	1	5.0%	6	30.0%
	SIE RelyX	4	20.0%	2	10.0%	2	10.0%
	APA Clearfil	1	5.0%	3	15.0%	8	40.0%
	APA RelyX	5	25.0%	3	15.0%	4	20.0%

P value = 0.25



GRAPH (1) Mean MSBS of different groups.



GRAPH (2) Compare failure types of different groups.

DISCUSSION

Several dopant agents have previously demonstrated the capability to infiltrate the grain boundary region of zirconia and exert high capillary and surface tension forces resulting in structural changes of the surface grains. SIE utilizes a specific glass infiltration agent that is able to diffuse in the grain boundaries and results in nano intergrain porosity. After rinsing off this agent, the surface of zirconia is readily capable of establishing a nano-mechanical bond with the adhesive resin composite of choice [(23)].

Low bond strength observed in sintered specimens has also been reported in other studies [(24-25)] using different combinations of surface treatment and bonding resin composite. Such findings indicate that establishing a strong chemical bond with zirconia is a difficult procedure for the MDP resin composite. These results could be further clarified by subjecting the specimens to artificial aging (e.g. thermocycling and long water storage) which is the aim for future studies [(24-25)].

The scanning electron microscopy (Fig.3-4) revealed that selective infiltration etching resulted in the creation of highly retentive surface capable of bonding with adhesive resin. SIE can also have left chemically reactive islets on the surface of the specimen and could thus have chemically modified the surface of zirconia to enable a better reaction with the adhesives and this agree with other studies [(1-26)].

In the current laboratory study, zirconia disks were bonded to resin composite disks (instead of dentin) because clinically, the weakest interface is observed to be between zirconia and resin composite cement [(27-28)].Therefore, the failure will be adhesive at zirconia and not at the dentin surface, meaning that adhesive on zirconia is less reliable than that dentin surface.

The microshear bond strength test, which has more advantages than shear bond strength SBS test was used because it allows testing small areas and make a precise map of tested surface[(29)]. This method also shows straightforward specimen preparation and yields precise results with small standard deviations [(30-31)].

Interestingly scanning electron microscopy revealed that selective infiltration etching resulted in the creation of a highly retentive surface capable of bonding with the adhesive resin of choice. The SIE method can also have left chemically reactive islets on the surface of the specimens and could thus have chemically modified the surface of zirconia to enable a better reaction with the primers. Further research work is needed to clarify this interesting issue. Once the resin-composite infiltrates the 3D inter-grain porosities, it becomes structurally integrated with the surface and higher forces are required to disrupt this bond[(1)].

In comparison, the microtensile bond strength test is cumbersome, technique sensitive and requires careful handling of fragile fragments.Trimming of specimens is an important step when preparing fragile specimens [(32)].

In this study short term water storage might be considered as one of limitations because long term water storage will deteriorate the bond and affect the data to a greater extent.

Previous results suggested that resin-zirconia bond strength was affected by different surface treatments and type of cement which was consistent with previous studies [(33-34-35-36-37)].The best adhesion strength was achieved with Clearfil. We suggest that use of cements containing 10-MDP monomers is an important factor in achieving durable adhesion to zirconia.

In contast, when Rely X cement was applied on a non treated surface, only inferior results was observed .

In terms of adhesion (bond) strength, it is difficult to define what is sufficient from clinical perspective this is under continuous academic debate. For example, 20 MPa has been widely considered clinically sufficient for durable resin-zirconia adhesion [38].

In the current study ,only samples cemented with Clearfil on either APA or SIE treated zirconia surfaces provided acceptable adhesive strength (21.67 and 26.18 Mpa).This confirms the influence of MDP monomer for achieving strong adhesion to zirconia(consistent with previous study) [39-40-41].

The use of Clearfil cement in the polished group could not provide a strong and durable bond only 6 MPa. Therefore as reported previously, mechanically creating a retentive surface is an important prerequisite for achieving strong and durable adhesion. Oyague et al.[40] and Miragaya et al.[41] reported that high bond strength were obtained by using 10 MDP monomer for achieving strong adhesion to zirconia.

Several laboratory studies have shown that APA cleanses the surface and increases surface roughness to improve micro-mechanical interlocking [42], as well as promotes chemical reaction of organophosphate groups in 10 MDP containing cements [43].

Significant differences in bond strength values between groups that underwent surface treatment and control group might be attributed to penetration of resin composite cement to rough surface of zirconia that facilitated nano or micromechanical interlocking of resin to zirconia [15-42].

In this study, SEM revealed that SIE technique created a highly retentive surface. This improved nanomechanical retention confirmed by a higher adhesion strength of 26.18 MPa for specimens which received SIE treatment, and only 9.1 MPa was found for polished specimens bonded with same adhesive resin (Clearfil).

The atomic force microscopic analysis by Casucci et al. [44] also revealed a significant improvement in average surface roughness with no signs of ceramic degradation. They also reported that surface available for bonding and presence of retentive spaces make this treatment promising for conditioning zirconia

Type of failure mode analysis basically showed adhesive failure when Rely X was used, it failed to bond strongly to zirconia. This could be related to absence of reactive functional group 10 MDP or their poor wettability due to high viscosity. When using Clearfil, the failure mode was predominantly cohesive with APA or SIE surface treatment .Previous analysis showed that higher mean MSBS was associated with higher percentages of cohesive failure in resin cement, which is consistent with the results of Yang[45]. More investigations in translucent zirconia should be carried out using other tests to address the controversy behind surface treatment.

Limitations of this study is the short period of water storage used and zirconia was bonded to composite disks instead of dentine.

According to results of this study, null hypotheses 1 and 2 were rejected as 10-MDP and SIE showed significant superior results.

CONCLUSIONS

SIE and APA in combination with 10 MDP containing resin composite cement established strong and durable adhesion to zirconia.

Rely X cement merely did not react with zirconia surface and is not recommended for adhesion to zirconia.

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